Development of Multilayer Analyzer Array Detectors for X–ray Fluorescence at the Third Generation Synchrotron Source

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Abstract. The development of Multilayer Analyzer Array Detector (MAAD) for X-ray fluorescence eliminates the count rate limitation encountered with multi-element Ge detectors. A 24-element multilayer detector has been fabricated that is tunable in a large energy region. This detector has been operational for more than two years at the BioCAT Beamline of the Advanced Photon Source at Argonne National Laboratory. Here we report our recent progress in developing multilayer detectors working in lower energy regions, in particular, performance at Ca Kα fluorescence energy and test results at soft x-ray energies. The band width of the analyzer response is found to be 3-4% of the fluorescence energy. Namely, at the Ca Kα energy, the band width is 140 eV; it is reduced to about 60 eV at Al Kα fluorescence energy. The throughput of the detector in this energy region (1.5-3.6 KeV) is 20% to 30%. These results demonstrate the feasibility for constructing multilayer analyzer array detectors for use in the soft x-ray region.

Introduction. It has been a concern that the multi-element Ge detector, which is widely used for biological XAFS data collection, is no longer adequate due to its count rate limitations when working at third generation synchrotron sources [1]. This concern has led to the proposal for the construction of fluorescence detectors using synthetic multilayers; this work was started at the Biostructures PRT beamline (X9) at Brookhaven National Laboratory, and continued at the BioPhysics Collaborative Access Team (BioCAT) project at the Advanced Photon Source, Argonne National Laboratory. Using linearly graded multilayers, the detector can be made tunable in a large energy region, which is an advantage compared to the earlier work on analyzer type detectors [2]. A 24-element multilayer analyzer array detector was commissioned in 2000 and has been extensively used at the BioCAT beamline for x-ray fluorescence data collection. The detector was designed to cover an energy region from 4 to 9 KeV and a solid angle of 2.5% of 4π at Ca K_α fluorescence energy. The design of the detector and its performance at various fluorescence energies will be the topic of another paper [3]. Here, we report the performance of the MAAD tested at Ca K_α energy, and preliminary testing results of the multilayers down to the 1-2 KeV energy region.

Results. Figure 1 shows the multilayer analyzer array detector [3]. Two motor assemblies are used to control the detector; one adjusts the entrance slit size and the other adjusts the orientation of the multilayer array. Two large area plastic scintillator/photomultiplier (PMT) detectors are used to collect fluorescence behind the multilayer array. The PMTs are generally operated under photon counting mode to reduce noise. The detector set up involves the position alignment of the detector relative to the beam and energy calibration with a fluorescence energy of particular interest [4].

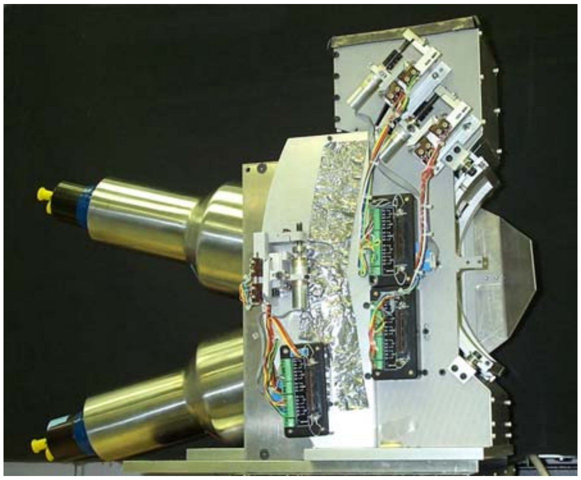

Figure 1. Photo of the multilayer analyzer array detector as reported in Ref. 3.

Figure 2 shows the detector calibration around Ca K_α energy on a 10 mM Ca solution sample. With x-ray energy set at 4250 eV and a focused beam of 0.3 mm vertical and 0.5 mm horizontal, the detector calibration observed K_α and K_β fluorescence peaks from the sample, as well as the peak for scattering photons. The energy resolution, taken as the full-width at half maximum (FWHM) of the peak, is approximately 140 eV.

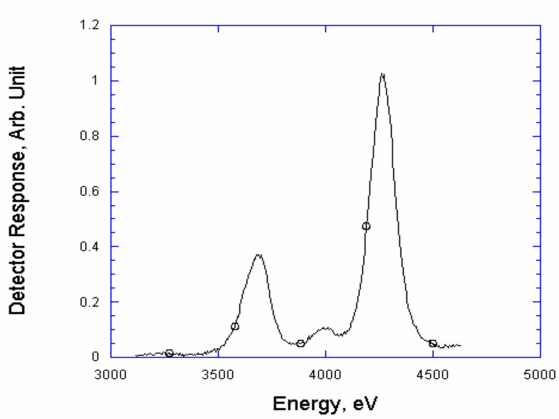

Figure 2. Detector calibration on a 10 mM Ca solution sample. The relative orientation of the multilayer elements has been converted to energy. The x-ray was set at 4250 eV.

The detector calibration was also performed on a concentrated Ca sample to measure the throughput [4] (data not shown). As expected [3], the throughput of the detector is approximately 30% at this energy.

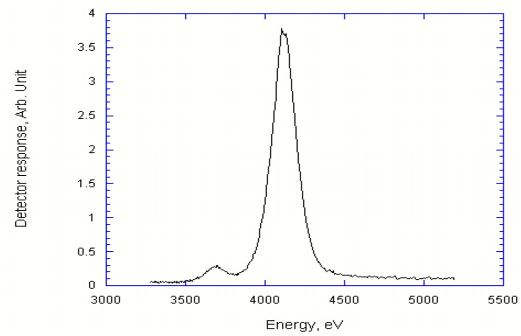

Figure 3. Detector calibration on a 1 mM Ca solution sample. The energy was set at 4200 eV.

The calibration was also performed on a 1 mM Ca solution sample (Figure 3). In this case, the K_α fluorescence is about 5% of the big scattering peak. The K_β fluorescence peak is invisible, buried by the large scattering peak. By setting the detector at the K_α fluorescence energy, XANES spectra were collected with a scan time of 50 seconds.

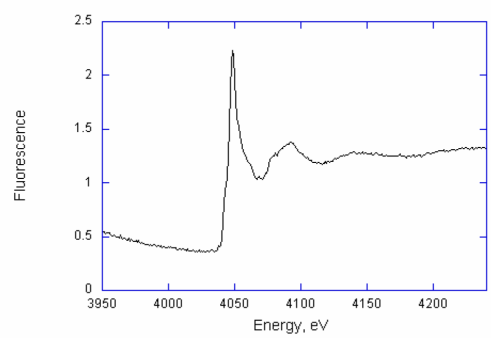

Figure 4. XANES spectra collected using the MAAD on the 1 mM Ca solution sample. The total data collection time is roughly 13 minutes.

Figure 4 shows the sum of these scans with a total data collection time of 15 minutes. As can be seen, the edge jump to background ratio is roughly 2 to 1. This gives a background rejection rate, which is defined as the ratio of signal to background with the detector to that without the detector, at roughly 40 times.

Based on the performance of MAAD at the Ca fluorescence energy, it is a logical step to perform feasibility tests of multilayers in the soft x-ray regime. Since the energy resolution of the multilayer analyzer detector is generally proportional to the energy, it is possible to get the energy resolution bellow 100 eV when working at these energies. Thus, we have carried out a preliminary experiment to examine the performance of multilayers at even lower energies at beamline X-19A of National Synchrotron Light Source (NSLS), Brookhaven National Laboratory. Using a Si (111) double crystal monochromator, the incident energy of the beamline was set at 2.4 KeV. A helium bag, which contains the multilayer analyzer, was attached to the helium sample chamber of the beamline. With the multilayer placed at a fixed distance from a sample, we measured the rocking curve of the multilayer by rotating it in the diffraction plane with fixed entrance slits. The signal after the multilayer was collected using a ZnS(Ag) screen coupled with a large area PMT. We first evaluated the rocking curve of an Al foil, and obtained a throughput of 23% (data not shown). We then measured the rocking curve for a sample of mixed Al and Si , which have their K_α fluorescence at 1478eV and 1740 eV, respectively (Figure 5).

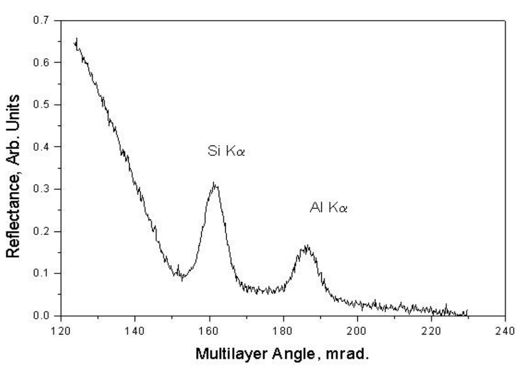

Figure 5. Tests of multilayers on a mixed system of Si and Al. The rocking curve is obtained by rotating the multilayer orientation in the diffraction plane with a fixed entrance window. The x-ray beam is set at 2.4 KeV with a vertical dimension of 0.5 mm.

As shown in the figure, both Si and Al fluorescence peaks are present at about 162 milli-radian and 186 milli-radian, respectively. The FWHM of the peaks is roughly 4%.

Discussion. We have shown experimentally that the width of the "rocking curve" from the graded multilayer as an analyzer is roughly 3-4%. This results in a band width of 140 eV at the Ca K_α fluorescence energy and approximately 60 eV at the Al Kα energy. The band width will be even smaller when the fluorescence energy is further lowered. This is an advantage compared to the energy resolution of the Ge detector at lower fluorescence energies. It has been reported that the energy resolution of the Ge detector can reach 130 eV at Mn Kα fluorescence energy with a solid state detector and digital signal processing [5]. However, the pulse peaking time needed is over 10 μs. This will further slow down the detector making it less efficient for working at the third generation sources. It should be emphasis here that, unlike the solid state detectors, the counting system of the MAAD sees only the good counts since the multilayer elements "select only" fluorescence photons. When operating with pulse counting mode of the scintillator/PMT detector, the maximum count rate can be as high as 10 million for a two PMT configuration [3]. The PMT channels can be easily added from 2 to 18, increasing the count rate to 10^8 photons/second. With a rejection rate at 30 to 50 times, the MAAD in practice has no count rate limitations.

The purpose of the multilayer detector project is to develop generally used x-ray fluorescence detectors with reasonable solid angle and good energy resolution for the research community using x-ray spectroscopy. The first unit is optimized to work from 4 to 9 KeV, in order to cover all biologically relevant transition metal elements [3]. The detector tested at the Ca K_α energy, shows an energy resolution of 140eV with a 30% throughput. The energy resolution can be further improved to about 60 eV when working at Al K_α fluorescence energy. The testing result indicates that the detector can work with better energy resolution with reasonable throughput at even lower energies. Therefore, we are fabricating a prototype multilayer analyzer array detector optimized for working bellow 2 KeV.

ACKNOWLEDGEMENTS.

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